



Total Phosphorus, USEPA in Persulfate Digests by Segmented Flow Analysis (SFA)

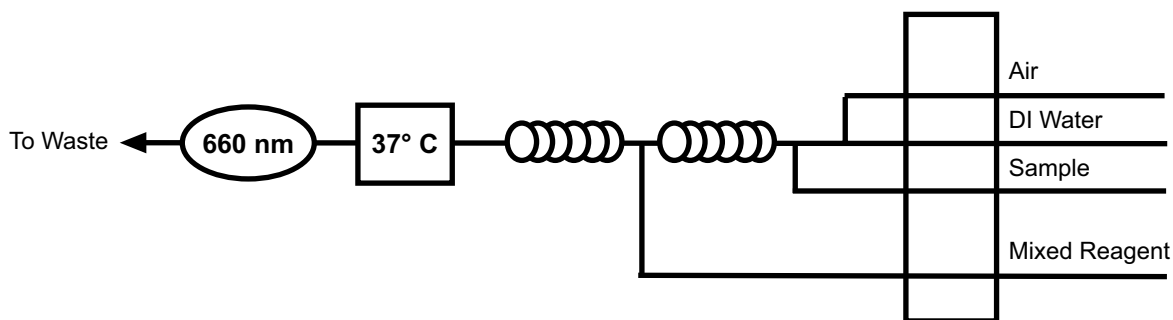
(Cartridge Part #A002596)

1.0 Scope and Application

- 1.1 This method is used for the determination of total phosphorus in drinking, surface and saline waters, domestic and industrial waste according to USEPA Method 365.1 (Reference 15.4).
- 1.2 The Method Detection Limit (MDL) of this method is 0.002 mg/L as phosphorus. The applicable range of this method is 0.003 to 10.0 mg/L phosphorus. The range may be extended to analyze higher concentrations by sample dilution.

2.0 Summary of Method

- 2.1 Prior to analysis, samples are digested using a persulfate digest to hydrolyze phosphorus to orthophosphate. The acidic digestate is then analyzed for orthophosphate.
- 2.2 Orthophosphate reacts with molybdenum (VI) and antimony (III) in an acid medium to form an anti-mony-phosphomolybdate complex. This complex is subsequently reduced with ascorbic acid to form a blue color, and the absorbance is measured at 660 nm (Reference 15.4).
- 2.3 The quality of the analysis is assured through reproducible calibration and testing of the Segmented Flow Analysis (SFA) system.
- 2.3 A general flow diagram of the SFA system is shown below (see Section 17.0 for a detailed flow diagram).



3.0 Definitions

Definitions for terms used in this method are provided in Section 16.0, "Glossary of Definitions and Purposes."

4.0 Contaminations and Interferences

- 4.1 Turbid samples may interfere with the photometric detector's ability to measure the true absorbance of the sample. Turbid samples should be filtered prior to analysis to reduce the possibility of such bias.
- 4.2 Iron, copper, and other metals may interfere with the accurate analysis of total phosphorus by binding with the orthophosphate in the sample, thus blocking the color formation reaction. Ethylene diaminetetraacetic acid (EDTA) is used in the buffer solution to eliminate this interference. Ferric iron of 50 mg/L or less, copper of 10 mg/L or less, and silica of 10 mg/L or less do not interfere (Reference 15.4).

5.0 Safety

- 5.1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been fully established. Each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level.
- 5.2 For reference purposes, a file of Material Safety Data Sheets (MSDS) for each chemical used in this method should be available to all personnel involved in this chemical analysis. The preparation of a formal safety plan is also advisable.
- 5.3 The following chemicals used in this method may be highly toxic or hazardous and should be handled with extreme caution at all times. Consult the appropriate MSDS before handling.
 - 5.3.1 Ammonium Molybdate, tetrahydrate $((\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O})$
 - 5.3.2 Ammonium Persulfate $((\text{NH}_4)_2\text{S}_2\text{O}_8)$
 - 5.3.3 Antimony Potassium Tartrate, hemihydrate $(\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6 \cdot \frac{1}{2}\text{H}_2\text{O})$
 - 5.3.4 Ascorbic Acid $(\text{C}_6\text{H}_8\text{O}_6)$
 - 5.3.5 Sulfuric Acid, concentrated (H_2SO_4)
- 5.4 Unknown samples may be potentially hazardous and should be handled with extreme caution at all times.
- 5.5 Proper personal protective equipment (PPE) should be used when handling or working in the presence of chemicals.

- 5.6 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

6.0 Apparatus, Equipment, and Supplies

- 6.1 Segmented Flow Analysis (SFA) System (OI Analytical Flow Solution IV) consisting of the following:
- 6.1.1 Model 502 Multichannel Peristaltic Pump
 - 6.1.2 Random Access (RA) Autosampler
 - 6.1.3 Expanded Range (ER) Photometric Detector with 5-mm path length flowcell and 660-nm optical filter
 - 6.1.4 Data Acquisition System (PC or Notebook PC) with WinFLOW™ software
 - 6.1.5 Orthophosphate/Total Phosphorus, USEPA Cartridge (OI Analytical Part #A002596)
- 6.2 Sampling equipment—Sample bottle, amber glass, with polytetrafluoroethylene (PTFE)-lined cap. Clean by washing with detergent and water, rinsing with two aliquots of reagent water, and drying by baking at 110°–150°C for a minimum of one hour.
- 6.3 Standard laboratory equipment including volumetric flasks, pipettes, syringes, etc. should all be cleaned, rinsed and dried per bottle cleaning procedure in Section 6.2.

7.0 Reagents and Calibrants

- 7.1 Raw Materials
- 7.1.1 Ammonium Molybdate, tetrahydrate ((NH₄)₆Mo₇O₂₄•4H₂O)
 - 7.1.2 Ammonium Persulfate ((NH₄)₂S₂O₈)
 - 7.1.3 Antimony Potassium Tartrate, hemihydrate (K(SbO)C₄H₄O₆•½H₂O)
 - 7.1.4 Ascorbic Acid (C₆H₈O₆)
 - 7.1.5 Dowfax® 2A1 (OI Analytical Part #A000080)
 - 7.1.6 Potassium Dihydrogen Phosphate (KH₂PO₄)
 - 7.1.7 Sulfuric Acid, concentrated (H₂SO₄)